G. Life Prediction of Diesel Engine Components

H. T. Lin, T. P. Kirkland, and A. A. Wereszczak
Oak Ridge National Laboratory
P.O. Box 2008, MS-6068
Oak Ridge, TN 37831-6068
(865) 576-8857; fax: (865) 475-6098; e-mail: linh@ornl.gov

Jeremy Trethewey and Craig Habeger
Caterpillar, Inc.
Peoria, IL 61656-1875
(309) 578-0056; fax: (309) 578-2953; e-mail: trethewey_jeremy@cat.com

DOE Technology Development Area Specialist: James J. Eberhardt
(202) 586-9837; fax: (202) 586-1600; e-mail: james.eberhardt@ee.doe.gov
ORNL Technical Advisor: D. Ray Johnson
(865) 576-6832; fax: (865) 574-6098; e-mail: johnsondr@ornl.gov

---

Contractor: Oak Ridge National Laboratory, Oak Ridge, Tennessee
Contract No.: DE-AC05-00OR22725
Subcontractor: Caterpillar, Inc., Peoria, Illinois

Objective

- Generate a database and characterize damage mechanisms of candidate advanced ceramics and intermetallic alloys.
- Apply and verify probabilistic life prediction and component design and verification methods for advanced diesel engine components.

Approach

- Evaluate the dynamic fatigue, rotary bending fatigue, and high-temperature fatigue and creep performance of candidate silicon nitride ceramics and TiAl alloys at elevated temperatures in air before and after long-term exposure to simulated engine environments, as well as engine field tests.
- Characterize the evolution and role of damage mechanisms, and changes in microstructure and chemistry, linked to the long-term mechanical performance and reliability of ceramics and intermetallic alloys.
- Predict the failure probability and reliability of complex-shaped diesel engine components subjected to application conditions via the use of life prediction codes.

Accomplishments

- Completed development of a dynamic fatigue database at elevated temperatures up to 1000°C in air for a commercial-grade silicon nitride, SN147-31E, acquired from Ceradyne, Inc.
- Completed characterization of the mechanical strength and microstructure for 16 prototype NT551 silicon nitride exhaust valves, as well as half-cylindrical valve stems, after a 500-h bench rig test at Caterpillar.
- Completed dynamic fatigue studies for diesel particulate filter (DPF) material at elevated temperatures in air.

Future Direction

- Characterize the retained mechanical properties and microstructure of Kyocera SN235P silicon nitride and TiAl exhaust valves designed for a Caterpillar natural gas engine after a field test, and verify the probabilistic component design and life prediction.
- Develop a static fatigue performance database at elevated temperature for specimens extracted from DPF substrates for long-term mechanical reliability and life prediction.
- Develop a high-temperature tensile creep and step-stress rupture database of TiAl alloys for probabilistic component design and life prediction for diesel exhaust valve and turbine wheel component application.
- Develop a mechanical database for biaxial discs extracted from TiAl turbine wheel airfoils to verify probabilistic component design and life prediction.

Introduction

There has been considerable interest in the potential for extensive use of advanced ceramics and intermetallic alloys in advanced diesel engine systems because of their superior thermomechanical properties at elevated temperatures. The implementation of components fabricated from these advanced materials would lead to significant improvement in engine efficiency and long-term durability and reduced nitrogen oxides (NO_x) and CO exhaust emissions as required in the 21st Century Truck Program. Interest has focused primarily on research into characterization and design methodology development (life prediction) for advanced silicon nitride ceramics and TiAl alloys to enable the manufacture of consistent, reliable complex-shaped components for diesel engine. The valid prediction of mechanical reliability and service life is a prerequisite for successful use of these materials in internal combustion engine components.

This research project has three primary goals: the generation of a mechanical engineering database, from ambient to high temperatures, of candidate advanced materials before and after exposure to simulated engine environments; the microstructural characterization of failure phenomena in these advanced materials and in components fabricated from them; and the application and verification of probabilistic life prediction methods using diesel engine components as test cases. For all three stages, results will be provided to both material suppliers and component end-users for use in refining and optimizing processing parameters to achieve consistent mechanical reliability, and in validating the probabilistic design and life prediction of engine components made from these advanced materials.

Approach

All flexural test specimens were longitudinally or transversely machined according to ASTM standard C1161 from production billets purchased from material suppliers and diesel engine components acquired from end-users.[1] Flexure testing was conducted in ambient air in a 4-point-bending apparatus using 20/40-mm, α-SiC, semi-articulating fixtures at temperatures ranging from 20 to 1000°C and at stressing rates of 30 MPa/s and 0.003 MPa/s. The 30-MPa/s test condition was chosen to evaluate the inert characteristic strength as a function of temperature, while the 0.003-MPa/s test condition was chosen to measure the change in slow crack growth (SCG) susceptibility at elevated temperatures. The dynamic fatigue tests were carried out per ASTM C1465 (Ref. 2). Pneumatic actuators were programmed with a PC to produce the desired loading rate (and corresponding stressing rate). Load was continuously measured as a function of time, and flexure strength was calculated using ASTM C1161. The accumulated strength data were then further analyzed. The strengths for each test set were fit to a two-parameter Weibull distribution using the program CERAMIC,[3] which uses maximum likelihood estimation as advocated in ASTM C1239 (Ref. 4). Reported results are uncensored because fractography analysis was not conducted in detail to identify strength-limiting flaws for all of the bend bars tested. Following the dynamic fatigue test, both optical and scanning electron microscope analysis were carried out on fracture surfaces and polished cross-sections of selected bend bars to characterize the fracture and degradation mechanisms. X-ray analysis was also carried out to evaluate the possible phase changes resulting from oxidation during dynamic fatigue tests or after long-term exposure to simulated engine environments, which could possibly cause degradation in mechanical performance and reliability.

Results

Dynamic Fatigue Response of Ceradyne SN147-E Silicon Nitride

Studies were completed of the dynamic fatigue behavior of a commercial-grade silicon nitride—SN147-31E, manufactured by Ceradyne Advanced Ceramic, Inc., CA—at temperatures of up to 1000°C in air. The SN147-31E has been processed with oxide sintering additives (i.e., Al_2O_3 and Y_2O_3) and contains a crystalline secondary phase achieved by proprietary post–heat treatment. The SN147-31E silicon nitride test specimens were longitudinally and transversely machined per the revised ASTM C1161 standard with a 600-grit surface finish.[1] The dynamic fatigue tests were carried out at 20 and 850°C and at stressing rates of 30 and 0.003 MPa/s in air per ASTM C1465 (Ref. 2). The 30 MPa/s rate is used to evaluate the inert characteristic strength as a function of temperature, and 0.003 MPa/s is applied to measure the slow crack growth (SCG) susceptibility at temperatures. Limited samples were also tested at 1000°C and at 30 MPa/s to evaluate high-temperature application limits and changes in life-limiting processes. The upper temperature limit in a diesel engine environment is about 850°C.

Dynamic fatigue results at 30 MPa/s showed that the transversely machined SN147-31E silicon nitride exhibited little or no decrease in characteristic strength at temperatures up to 1000°C compared with those obtained at 20°C (as shown in Table 1).

Also, the SN147-31E samples exhibited higher Weibull moduli at elevated temperatures than at 20°C, indicative of a change in strength-limiting flaws as a function of test temperature. On the other hand, mechanical results at 850°C indicated that there is an apparent decrease in characteristic strengths (about 19%) when tested at 0.003 MPa/s. In addition, the fatigue exponent of transversely machined SN147-31E decreased from 338 at 20°C to 39 at 850°C, indicative of an increased susceptibility to SCG processes at high temperature (Figure 1). The decrease in dynamic fatigue exponent value at elevated temperatures could be attributed to the change in material state (e.g., softening of secondary phase), as seen in the case for the GS44 silicon nitride.[5] The low fatigue exponent obtained for SN147-31E at elevated temperatures suggests that the secondary phase was not

Table 1. Summary of uncensored Weibull and strength distributions for Ceradyne SN147-31E silicon nitride specimens longitudinally and transversely machined per ASTM C1161. Data for SN147-31N machined longitudinally as well as transversely are used for reference

Material	Number of specimens tested	Stressing rate (MPa/s)	Temperature (°C)	Uncens. Weibull modulus	± 95% uncens. Weibull modulus	Uncens. char. strength (MPa)	± 95% uncens. char. strength (MPa)
SN147-31N-Long	15	30	20	21.73	14.07. 31.09	836	814, 858
SN147-31N-Long	15	30	850	20.35	13.58, 28.20	777	755, 799
SN147-31N-Long	15	0.003	850	16.19	10.57, 23.02	732	706, 757
SN147-31N-Trans	15	30	20	13.76	8.96, 19.59	677	649, 705
SN147-31N-Trans	15	30	850	18.26	11.67, 26.47	639	619, 659
SN147-31N-Trans	15	0.003	850	19.95	12.83, 28.63	620	602, 638
SN147-31E-Long	15	30	20	17.30	10.86, 25.58	668	645, 690
SN147-31E-Long	15	0.003	20	16.19	10.60, 22.98	620	598, 642
SN147-31E-Long	15	30	850	9.36	6.01, 13.62	604	567, 641
SN147-31E-Long	15	0.003	850	12.06	7.70, 17.52	509	485, 533
SN147-31E-Long	15	30	1000	16.49	10.61, 23.66	585	564, 605
SN147-31E-Trans	15	30	20	7.44	4.89, 10.58	623	575, 671
SN147-31E-Trans	15	0.003	20	8.99	5.61, 13.45	607	568, 646
SN147-31E-Trans	15	30	850	43.94	28.37, 62.78	642	634, 651
SN147-31E-Trans	15	0.003	850	18.02	11.93, 25.18	521	505, 538
SN147-31E-Trans	15	30	1000	34.66	21.97, 50.70	602	591, 611

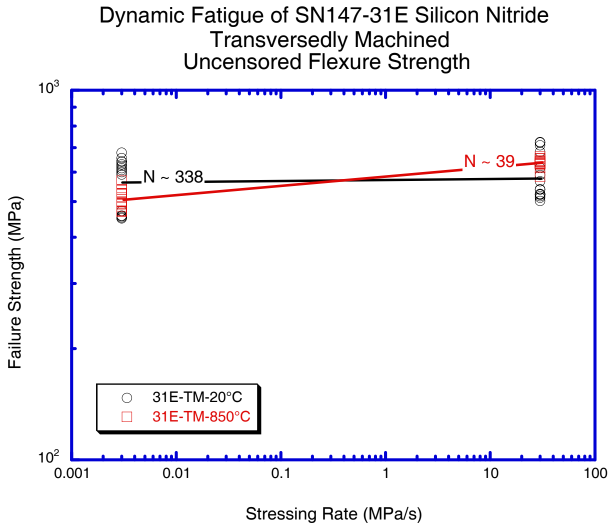

Figure 1. Failure strength versus stressing rate curve of SN147-31E transversely machined and tested at 20 and 850°C in air.

completely crystallized, and thus the post–heat treatment procedure needs to be refined to ensure high-temperature mechanical performance and reliability.

Mechanical Characterization of NT551 Silicon Nitride Exhaust Valves

Mechanical strength testing was conducted on 16 NT551 silicon nitride valves (14 with 30° and 4 with 45° valve seat angles) after testing on a 500-h bending rig at Caterpillar. In general, optical microscopy examinations showed that no surface damage or flaws were apparent after the bench rig test. However, there were distinct valve seat markings at the valve and stainless steel valve seat contact point. Strength testing was carried out at room temperature, with a compression text figure specially designed in-house, to evaluate the effects of a 500-h bench rig test on mechanical performance (Figure 2). The test results of as-received NT551 valves obtained using a hydraulic chamber facility were used as a baseline for comparison (Figure 3). Mechanical results for the valves tested at 500 h show that the characteristic strength and the Weibull strength distribution were similar to those obtained for the as-machined valves, suggesting no surface/subsurface damage was introduced during the bench rig test (Figure 4). Note that nondestructive evaluation carried out at Argonne National Laboratory on the valves tested at 500 h also indicated no apparent damage/flaws, consistent with the present mechanical testing results.

Studies were also conducted of the mechanical properties of NT551 silicon nitride valve stems after a 500-h bench rig test. The objective was to see whether the 500-h bench rig test would introduce any surface damage to the valve stem surfaces and thus degrade the mechanical strength. The half-cylindrical valve stems were machined and then

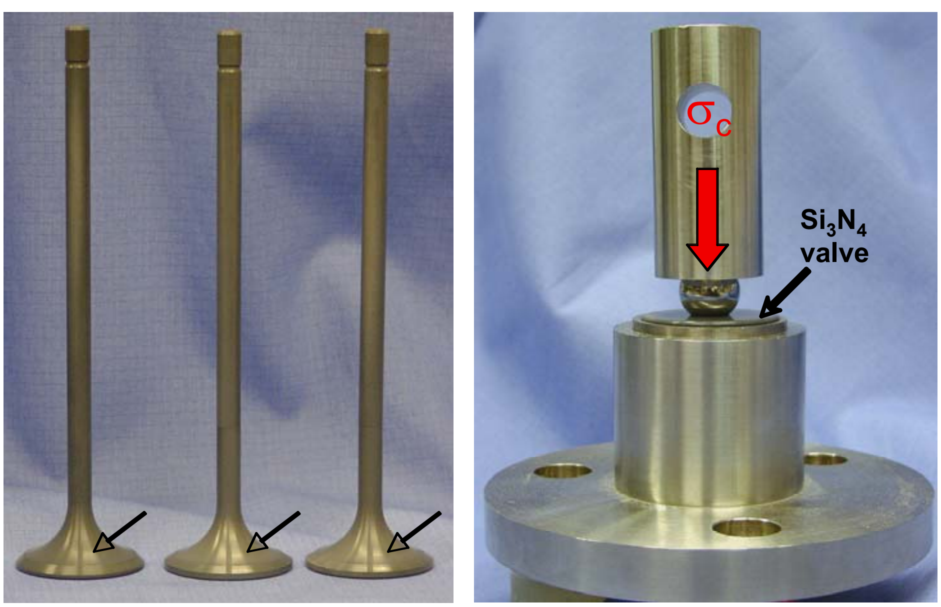

Figure 2. Photos of silicon nitride exhaust valves after testing on a 500-h bend rig at Caterpillar and on a mechanical testing fixture. The valve strength test was on a Universal Instron machine.

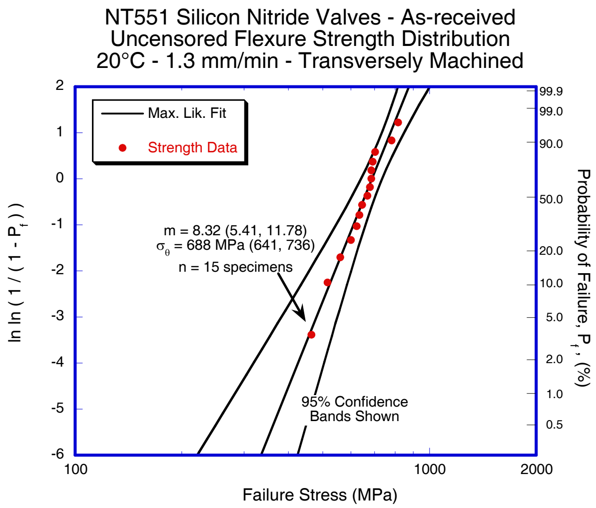

Figure 3. Strength distribution of as-received Norton NT551 silicon nitride valve transversely machined and tested at 20°C.

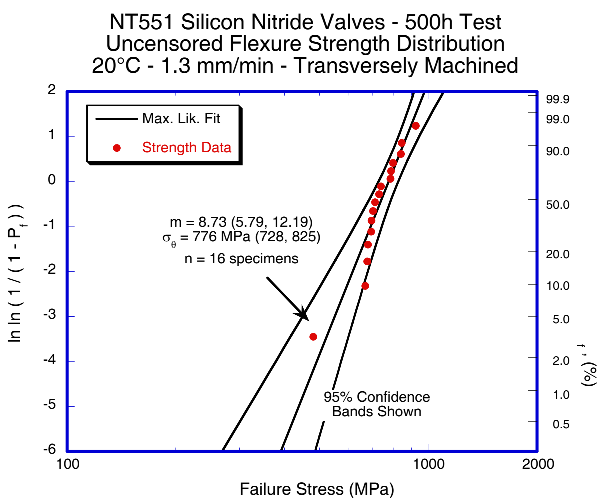

Figure 4. Strength distribution of Norton NT551 silicon nitride valve transversely machined and tested for 500 h at 20°C.

tested using a fixture with 30/60-mm spans with a 2-mm/min crosshead speed at room temperature. Figure 5 shows the uncensored Weibull strength distribution of these half-cylindrical valve stem specimens. The mechanical results show a characteristic strength of 1135 MPa and Weibull modulus of 12.7, comparable to and/or better than the as-machined cylindrical samples. The results indicate excellent wear resistance. The slight increase in characteristic strength might be due to

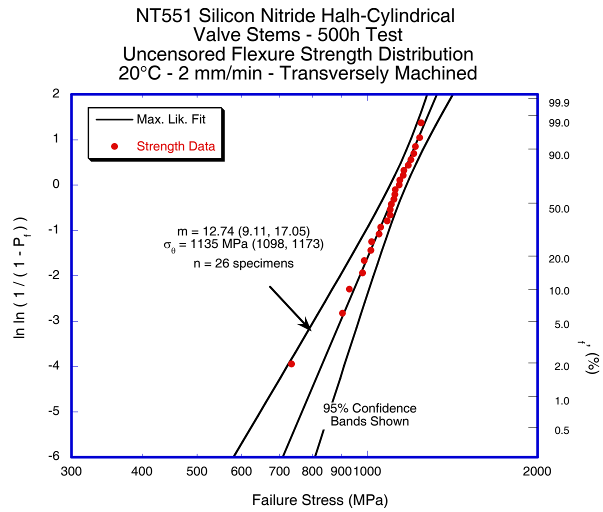

Figure 5. Strength distribution of Norton NT551 silicon nitride half-cylindrical valve stems transversely machined and tested for 500 h at 20°C.

the surface smoothing effect resulting from the contact between the silicon nitride valve stem and the valve guide.

Dynamic Fatigue Performance of Diesel Particulate Filters

Studies of the mechanical properties of a DPF substrate, especially dynamic fatigue behavior at elevated temperatures, were initiated and carried out during this fiscal year. The objective is to develop a mechanical database for predicting long-term reliability and durability. Two different types of samples, with 3×4 cells and 4×6 cells, were employed to evaluate how size influences the mechanical response and fracture process. Tests were carried out at temperatures of 600, 800, and 900°C at stressing rates of 5, 0.005, and 0.00005 MPa/s in air. The dynamic fatigue procedures carried out for DPF materials are similar to those employed for monolithic ceramic specimens (ASTM C1465). Figure 6 shows the preliminary dynamic fatigue results for DPF samples with 4 × 6 cells. The results showed that the DPF material exhibited similar low dynamic fatigue exponents (N ~ 40–44) in the temperature range employed. The low fatigue exponents obtained suggest that the DPF material might exhibit susceptibility to SCG processes at temperatures. More test specimens and a lower stressing rate need to be

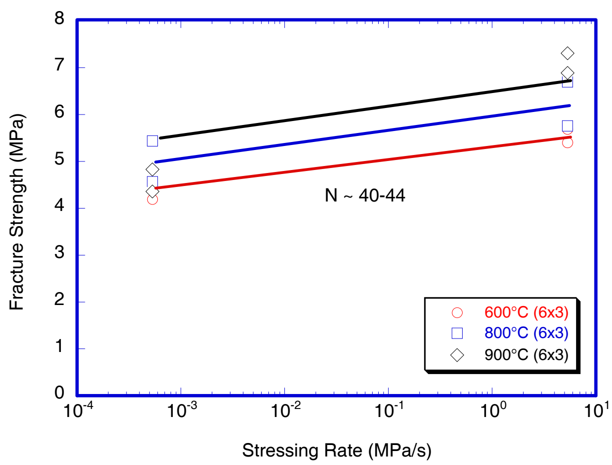

Figure 6. Fracture strength versus stressing rate of DPF material as a function of test temperature.

studied to provide a better understanding of long-term reliability and life-limiting processes.

References

1. "Standard Test Method for Flexural Strength of Advanced Ceramics at Ambient Temperatures," ASTM C1161, *Annual Book of ASTM Standards*, Vol. 15.01, American Society for Testing and Materials, West Conshohocken, Pennsylvania, 1999.

2. "Standard Test Method for Determination of Slow Crack Growth Parameters of Advanced Ceramics by Constant Stress-Rate Flexural Testing at Elevated Temperatures," ASTM C1465, *Annual Book of ASTM Standards*, Vol. 15.01, American Society for Testing and Materials, West Conshohocken, Pennsylvania, 1999.

3. *Life Prediction Methodology for Ceramic Components of Advanced Heat Engines, Phase 1*, ORNL/Sub/89-SC674/1-2, prepared by AlliedSignal Engines, DOE Office of Transportation Technologies, 1995.

4. "Practice for Reporting Uniaxial Strength Data and Estimating Weibull Distribution Parameters for Advanced Ceramics," ASTM C1239, *Annual Book of ASTM Standards*, Vol. 15.01, American Society for Testing and Materials, West Conshohocken, Pennsylvania, 1999.

5. A. A. Wereszczak, H. -T. Lin, T. P. Kirkland, M. J. Andrews, and S. K. Lee "Strength and Dynamic Fatigue of Silicon Nitride at Intermediate Temperatures," *J. Mater. Sci.*, **37**, 2669–2684 (2002).

Presentations and Publications

H. T. Lin, T. P. Kirkland, A. A. Wereszczak, and M. J. Andrews, "Strength Retention of Silicon Nitride After Long-term Oil Immersion Exposure," *J. Mater. Sci.* (in press).